PRODUCTION OF CONCENTRATES OF TITANIUM DIOXIDE IN POLYCAPROLACTAM

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ABSTRACT
Concentrates of from 20 to 50% by weight of titanium dioxide in polycaprolactam are produced by a process in which a 5-15% strength by weight aqueous suspension of titanium dioxide is metered into a solution of caprolactam in water at from 100° to 150° C. at the same rate at which water is distilled off, and after the water has been distilled off the resulting mixture is polymerized at from 240° to 280° C.

2 Claims, No Drawings
PRODUCTION OF CONCENTRATES OF TITANIUM DIOXIDE IN POLYCAPROLACTAM

It is frequently desired to produce polycaprolactam delustered with titanium dioxide, in order to spin from this material filaments which have a particularly low luster. For this purpose, it is necessary to incorporate finely divided titanium dioxide into the polymerizing caprolactam melt. The titanium dioxide is introduced into the polymerizing melt as a suspension in water or caprolactam, as disclosed in "Klare synthetische Fasern aus Polyamiden", Akademie-Verlag, Berlin, 1963, page 170. This procedure has the disadvantage that titanium dioxide is readily flocculated by the chain regulator present, leading to nodules in the spun filament. Moreover, the titanium dioxide suspensions used do not have a long shelf life; titanium dioxide is deposited, thus giving suspensions of varying concentration. Attempts have been made to eliminate this disadvantage by adding protective colloids. However, extraneous material is thereby introduced into the polyamide melt, and this is undesirable, particularly when polycaprolactam is used for fiber production.

It is an object of the present invention to provide concentrates of titanium dioxide, which have a long shelf life and a constant concentration, which can readily be dispersed in the caprolactam or polycaprolactam melt, and in addition, owing to the omission of water, do not adversely affect the capacity of the polymerization plant.

We have found that this object is achieved by a process for the production of concentrates containing from 20 to 50% by weight of titanium dioxide in polycaprolactam, wherein a 5-15% strength by weight aqueous suspension of titanium dioxide is metered into a solution of caprolactam in water at from 110° to 150° C. at the same rate at which water is distilled off, and after all the water has been distilled off the resulting mixture is polymerized at from 240° to 280° C. The resulting polymer thus obtained is extruded as strands and the extrudates are granulated. The aqueous titanium dioxide concentrates obtained according to the process of the invention are suitable for delustering polycaprolactam during its production.

The Example which follows illustrates the process according to the invention.

EXAMPLE

(a) Production of a titanium dioxide suspension

275 kg of titanium dioxide (anatase) of mean particle size 2 μm and 68 kg of caprolactam are worked for 2 hours at 20° C in a kneader. Thereafter, 60 kg of demineralized water and 0.1% by weight (based on titanium dioxide) of disodium phosphate .12H₂O are added, and the mixture is kneaded for a further 2 hours. The resulting 68% strength by weight paste is flushed with 2,000 l of demineralized water from the kneader through a turbomixer into a container, and is homogenized by circulating it for one hour. The mixture is left to stand for 48 hours for agglomerates and relatively large titanium particles to settle out, and thereafter the suspension is decanted from the titanium dioxide sediment. The resulting aqueous suspension contains about 11% by weight of titanium dioxide of particle size 0.2-0.9 μm.

(b) Production of a titanium dioxide concentrate

2,200 l of caprolactam and 350 l of demineralized water are introduced into a pressure-resistant vessel and heated to 130° C with stirring. A pressure of 1.5 bar develops. 9,000 kg of a 11% strength by weight titanium dioxide suspension prepared according to (a) are metered in over the course of 11 hours, and at the same time water is distilled off continuously. After all the titanium dioxide suspension has been added, the temperature rises to 150° C, thus indicating that the distillation of water has ceased. Thereafter, the mixture obtained, which still contains about 6% by weight (based on titanium dioxide) of water, is filtered through two double sieves of 17 μm mesh size. The filtered mixture is polymerized for 7.5 hours at 258° C under a pressure of...
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7 bar. The melt is then discharged in the form of strands, and these are granulated. The granules contain 31% by weight of titanium dioxide.

We claim:

1. A process for the production of a concentrate of from 20 to 50% by weight of titanium dioxide in poly-caprolactam, wherein a 5–15% strength by weight aqueous suspension of titanium dioxide is metered into a solution of caprolactam in water at from 110° to 150° C. at the same rate at which water is distilled off, and after all the water has been distilled off the resulting mixture is polymerized at from 240° to 280° C.

2. A process as claimed in claim 1, wherein the aqueous titanium dioxide suspension used has been obtained by kneading titanium dioxide powder with dry caprolactam, then kneading these materials with the addition of water, with or without the addition of a di-alkali metal phosphate, suspending the resulting paste in water, allowing the coarse titanium dioxide particles to settle out, and decanting the suspension from the sediment.